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(54) **Interference filters.**

(57) A thin film interference filter capable of transmitting a desired proportion of visible radiation while reflecting a large portion of incident solar radiation comprising :
a transparent substrate ;
a first dielectric layer ;
a first metal precoat layer ;
an optically reflective metal layer ;
a second metal precoat layer ; and
a second dielectric layer
in which one or both of the dielectric layers is a composite comprising silicon nitride and one or more of zirconium nitride, titanium nitride, and hafnium nitride or mixtures thereof.

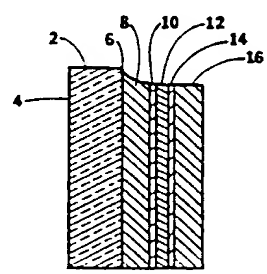

FIG._1A.

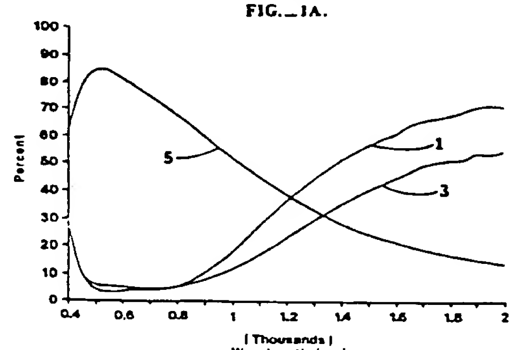

FIG._1B.

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This invention relates generally to visibly transparent infrared reflecting interference filters and, more particularly, to a durable low-emissivity thin film filter.

The use of transparent panels in buildings, vehicles and other structures for controlling solar radiation is quite prevalent today. The goal of solar control is to transmit light while excluding much of the solar energy, thus decreasing the amount of air condition or cooling required, and conserving energy. In addition, modified glass as a structural material provides the colour flexibility architects desire.

Various processes have been employed to alter the optical properties of these panels, including coating glass or plastic substrates by various techniques such as electrolysis, chemical vapour deposition and physical vapour deposition, including sputtering with planar magnetrons. For instance, thin metal films have been deposited on glass or plastic to increase the reflectance of solar radiation. Windows deposited with a multi-layer dielectric-metal-dielectric coating that exhibits high visible transmittance, and high reflectivity and low emissivity in the infrared range, are even more energy efficient. The index of refraction of the dielectric layer is preferably 2.0 or greater in order to minimise the visible reflectance and enhance the visible transmittance of the window. This dielectric layer which often consists of metal oxide coating also offers additional protection to the fragile metal films. The optical properties of panels can also be modified by altering the composition of the substrate material. Nevertheless, interference filter panels manufactured by the above-described methods have been only partially successful in reflecting solar radiation to the degree required for significant energy conservation.

The present invention is concerned with the provision of a durable, thin-film interference filter which transmits visible light while reflecting infrared radiation to render them useful in architectural panels which give less reflected colour of visible light over a wide band in particular.

The present invention provides a durable, thin-film interference filter which comprises a substrate onto which is deposited a dielectric layer, followed by at least one further metal and dielectric layers. In between each of the dielectric and metal layers is deposited a "nucleation" or glue layer that promotes adhesion between the dielectric to the metal. In one preferred embodiment of the invention the interference filter comprises a five layer structure wherein one or both of the dielectric layers is formed of a composite material containing zirconium nitride and silicon nitride. It has been found that mixing zirconium nitride with silicon nitride creates a composite layer that has a high refractive index and excellent transparency in the visible region. Moreover, the optical properties of this composite layer can be adjusted by varying the relative amounts of zirconium nitride and silicon nitride.

The dielectric layers of the inventive interferences filters can be reactively sputtered by a rotatable cylindrical magnetron. Composite layers can be formed by cosputtering from dual cathode targets or from one or more alloy targets. A feature of the inventive process is that by reducing the intrinsic stress of the second dielectric layer, an extremely hard and chemically resistant thin film coating is produced. In sputtering silicon nitride as the second dielectric layer, it was demonstrated that the intrinsic stress of this layer can be reduced by orienting the magnetic assembly of the cathode at an acute angle vis-à-vis the substrate.

For a better understanding of the invention, reference is now made, by way of exemplification only, to the accompanying drawings in which:

Figure 1a is a cross-sectional view of a five layer design thin-film interference filter produced in accordance with this invention.

Figure 1b is a graph illustrating the spectral transmittance and reflectance of a thin-film interference filter.

Figure 2 is a cross-sectional view of a cathode assembly.

Figure 3 is a graph illustrating the spectral transmission in the visible light region for a composite film.

Figure 4 is a graph illustrating the spectral reflection in the visible light region for a composite film.

Figure 5 is a graph illustrating the spectral adsorption in the visible light region for a composite film.

A thin-film interference filter incorporating the present invention is shown in Figure 1a. As shown therein, the filter consists of a transparent substrate 2 which is provided with two planar parallel surfaces 4 and 6, in which surface 4 is exposed to the medium and surface 6 is coated. The substrate can be formed of any suitable transparent material; however, the substrate is preferably a material which has superior structural properties and minimum absorption in the visible and near-infrared spectra regions where the solar energy is concentrated. Crystalline quartz, fused silica, soda-lime silicate glass, and plastics such as polycarbonates and acrylates, are all preferred substrate materials.

Deposited onto the substrate surface 6 is a first dielectric layer 8 that is preferably made of a material having an index of refraction of greater than about 2.0, and most preferably between 2.4 and 2.7. Suitable dielectric layer materials include metal oxides such as titanium oxide, tin oxide, zinc oxide, indium oxide (optionally doped with tin oxide), bismuth oxide, and zirconium oxide. Yet another suitable material is silicon nitride. In accordance with the invention, a particularly suitable dielectric material comprises a composite film containing zirconium nitride and silicon nitride (collectively referred to herein as "SiZrN") that may be fabricated by cosputtering from

dual targets or from a single alloy target of a DC cylindrical magnetron, as described herein.

Zirconium nitride is an electrically conductive material which has very good optical reflectance in the infrared spectrum; however, this material is very absorbing in the visible portion of the spectrum and cannot be used on devices requiring high transparency. Silicon nitride, on the other hand, is very transparent in the near UV through the near IR spectrum (350 nm, 2.0 microns). It was discovered that mixing zirconium nitride with the silicon nitride creates a composite film that has a high index of refraction (>2.10) and excellent transparency in the visible spectrum. The film also demonstrates good chemical and mechanical durability. Furthermore, by employing cosputtering with dual cathode targets, the index of refraction of the film can be adjusted by varying the amount of power to each cathode and/or the gases used in the process. The index of refraction of the film so fabricated ranges from approximately 2.00 to 2.45.

Besides SiZrN, composite films comprising titanium nitride and silicon nitride (collectively referred to herein as "SiTiN") or comprising hafnium nitride and silicon nitride (collectively referred to herein as "SiHfN") can also be used. SiTiN and SiHfN composite films can also be prepared by cosputtering from dual or single targets. Finally, a composite film comprising a mixture of silicon nitride, zirconium nitride, titanium nitride, and/or hafnium nitride can be used as the first dielectric layer. As will be described further below, the refractive index of the composite films will vary depending on the relative amounts of the different nitrides that comprise each film.

It has been found that when silicon nitride is used as the first dielectric layer, the visible light transmission of the inventive filter is slightly less than the transmission when titanium oxide or a composite film is used.

The thickness of the first dielectric layer ranges from approximately 200 to 500 Å, and more preferably from approximately 300 to 350 Å.

As shown in Fig. 1a, the inventive filter next comprises of a first metal precoat 10 that is deposited over the first dielectric layer. Precoat layer 10 is preferably maintained as thin as possible so that it will have very little, if any, adverse effect upon the optical characteristics of the filter or the subsequent metal laver. Precoat layers with thicknesses ranging from approximately 5 to 20 Å have been satisfactory; more preferably, the thickness is between approximately 8 to 16 Å. This thin precoat layer can be formed from any number of metals. It has been found that nickel-chromium alloy comprising approximately 1 to 80 percent nickel and approximately 1 to 20 percent chromium can be used as a precoat; more preferably, the alloy content is approximately 80 percent nickel and 20 percent chromium. Other metals and alloys thereof that can be used as a precoat include nickel, chromium, rhodium, platinum, tungsten, molybdenum, and tantalum. The precoat layer apparently acts as a glue or .Nucleation layer and as a stress reducing layer. It is believed that while the precoat layer is thin enough not to adversely affect the optical properties of the filter, it causes the metal film 12 to behave as if it were a homogeneous metal slab.

Next, a partially reflective metal layer 12 is deposited onto the first precoat layer. The metal layer reflects infrared-radiation, yet allows for sufficient visible light transmission. The metal layer can be formed from a number of materials, with silver being particularly satisfactory. Other metals which also can be utilised include gold, copper and platinum. The thickness of the metal layer ranges from approximately 40 to 150 Å, and more preferably, from approximately 90 to 110 Å.

In this preferred embodiment, a second metal precoat layer 14 is then deposited onto the metal layer which is followed by the final dielectric layer 16. This second metal precoat layer can be formed from the same material and in the same thickness range as precoat layer 10.

The second dielectric layer can be made of silicon nitride that is formed by reactive sputtering a cylindrical magnetron. This layer has a thickness from approximately 350 to 500 Å, and more preferably from approximately 450 to 475 Å. The above referenced composite films can also be used although the relative proportion of silicon nitride in each film should be adjusted so that the refractive index ranges preferably from approximately 2.04 to 2.10. When a composite film is used for the second dielectric layer, its thickness should be from approximately 300 to 500 Å preferably 350 to 375 Å. However, whether silicon nitride or a composite substance is used as the second dielectric layer, the layer most preferably exhibits low intrinsic stress as described further below. A suitable composite film is SiZrN comprising approximately 80-83% by weight silicon nitride and the balance zirconium nitride. This particular film has a refractive index of approximately 1.85 to 2.2. A preferred SiZrN composite film has a refractive index of about 2.08. As will be described below, the inventive filters offer excellent mechanical and corrosion resistance.

The precoat and metal layers were deposited with a D.C. planar magnetron. Other techniques including E-beam evaporation could have also been employed. The dielectric layers of the inventive filter were prepared by DC-reactive sputtering with a rotating cylindrical magnetron. The magnetron reactive sputtering technique is particularly useful for depositing dielectric films. While there are other techniques for depositing the dielectric layers such as thermal oxidation and LPCVD (low pressure chemical vapour deposition), these methods suffer from, among other things, slow deposition rates. Moreover, RF planar magnetron sputtering for depositing di-

electric material is impractical for large-scale industrial applications because of the enormous power requirements and RF radiation hazards. A description of a cylindrical magnetron suitable for depositing substrates with the dielectric materials is found in Wolfe et al., U.S. Patent 5,047,131, issued September 10, 1991, incorporated herein by reference. To provide additional protection to the inventive filter, a plastic laminate can be applied to the filter of Fig. 1a. See Young et al., U.S. Patent 4,965,121, issued October 23, 1990 incorporated herein by reference.

In fabricating the inventive filter, it was found that by reducing the intrinsic stress of the second dielectric layer 16, an extremely hard and chemically resistant thin film coating is produced.

Stress is an important variable that is inherent in each layer of a thin film stack. There are generally two stress states: (1) compressive, where the film is trying to expand on the substrate and, (2) tensile, where the film is trying to contract. In magnetron systems, the pressure of the vacuum depositing chamber is an important factor which influences stress. It is believed that at sufficiently low pressures, sputtered atoms and reflected neutral gas atoms impinge on the film at nearly normal incidence with high energy because at lower pressures there are fewer collisions within the plasma (larger mean free path). This mechanism, as reported by Hoffman and Thorton in Thin Solid Films. 40, 355 (1977), is known as "atomic peening", and is believed to cause compression in films.

At higher working pressures, the sputtered atoms collide with atoms in the plasma more frequently. Sputtered material reaches the substrate at oblique incidence and with lower energies. The decrease in kinetic energy of the incident atoms makes the peening mechanism inoperative. The decrease in the flux of atoms arriving at normal incidence results in "shadowing" -- voids remaining from the nucleation stage of film growth are not filled because nucleation sites shadow the obliquely arriving atoms. Shadowing and "competing cone growth" can lead to isolated columnar grain structures and an extensive void network. Messier and Yehoda, J. Appl. Phys., 58, 3739 (1985).

Whatever the cause of internal stress in sputtered films, there is, for a given set of system parameters (e.g., magnetron geometry, deposition rate, film thickness, gas pressure), an abrupt transition from compression to tension at a critical pressure which depends on the atomic mass of the material. (Hoffman and Thorton, Thin Solid Films, 45, 387 (1977); Hoffman and Thorton, J. Vac. Sci. Technol., 20, 355 (1982); Hoffman and Thorton, J. Vac. Sci. Technol., 17, 380 (1980).) Above this critical pressure, tensile stresses gradually decrease to zero. The relaxation of stress beyond some maximum tensile stress point was reported for chromium sputtered in argon and molybdenum sputtered in xenon. Shih et al., "Properties of Cr-N Films Produced by Reactive Sputtering", J. Vac. Sci. Technol. A4 (3), May/June 1986, 564-567.

In depositing silicon nitride as the second dielectric layer with a rotatable cylindrical magnetron, it was found that the intrinsic stress of the silicon nitride layer can be reduced by orienting the magnetic assembly of the cathode at an acute angle. As shown in Fig. 2, which is a cross-sectional view of cathode 20 and substrate 21, the magnetic assembly 18 has a "W" configuration with three elongated magnetics 24, 26, and 28. The permanent magnetics used formed an unbalanced system which. is typical for rotatable cylindrical magnetrons. As is apparent, the assembly is oriented at an acute angle α of approximately 45° so as to direct sputtered material towards the substrate 29 as it enters the deposition chamber. Angle α can range from approximately 30° to 80°. Silicon nitride layers so deposited have approximately one-fourth the intrinsic stress of silicon nitride layers produced when the assembly is at a normal angle relative to the substrate.

Experimental Results

A low-emissivity interference filter having the structure as shown in Fig. 1a comprising a glass substrate, a titanium oxide first dielectric layer, nickel-chromium alloy precoat layers, a silver metal layer, and a silicon nitride second dielectric layer was fabricated in an in-line magnetron system manufactured by the Applicants Airco Coating Technology Division. It is known that TiO_2 is the predominant form of titanium oxide created in the sputtering process. However, it is believed that other forms are produced as well. Thus, unless otherwise stated, TiO_2 will represent all forms of titanium oxide produced. The system comprises of five magnetrons arranged in series, with each magnetron depositing one of the five layers of the filter. The second, third, and fourth are planar magnetrons for depositing the first precoat, metal, and second precoat layers respectively. The planar magnetrons, each comprising of a model HRC-3000 unit, were manufactured by Airco Coating Technology. The first and fifth magnetrons are cylindrical magnetrons to deposit the dielectric layers. The cylindrical magnetrons, each comprised of a C-MagT model 3000 cathode, also manufactured by Airco Coating Technology.

The target(s) for each of the cylindrical magnetrons was conditioned using an inert gas, thereafter the process gas was added until the desired partial pressure was reached. The process was operated at that point until the process was stabilised. The substrate was then introduced to the coat zone of the first cylindrical mag-

netron and the film was applied. The substrate used was soda lime glass.

For depositing a first dielectric layer comprising titanium oxide, a C-MAG (Trade Mark) rotatable magnetron employing a titanium target was used. Alternatively, a planar magnetron can be employed. Argon was the inert gas and oxygen was the reactant gas. When depositing silicon nitride in the cylindrical magnetron, argon was used as an inert gas and nitrogen was used as the reactant gas. The partial pressure of the gas was determined by the transition from the nitride mode to the metallic mode. Experiments were run as close to that transition as practicable. The pressure and flow rate of the sputtering gases were controlled by conventional devices.

Because the electrical conductivity of pure silicon is so low that it is unsuitable for sputtering with direct current, the silicon target was impregnated or doped with a small amount of aluminium in the range of from 2-4%. The target was prepared by plasma spray. The sputtering source was connected to an appropriate direct current power source having provision for automatically maintaining the voltage, current or power, as desired. The magnet assembly of the single cathode was oriented at an angle of approximately 45° from normal.

With nitrogen as the sputtering gas, the coating contained a mixture of aluminium and silicon nitrides. All of these components are relatively hard and form an amorphous film that acts as a strong barrier. However, the amount of aluminium in the film did not interfere with formation of the desired silicon based compound films. In the course of the experiments, films were sent out for independent RBS (Rutherford Back-Scattering) sampling to determine the composition of the compound. The silicon nitride measured 42% Si/57% N, which is very close to the theoretical 3:4 ratio for nitride (Si_3N_4).

Table 1 sets forth the process data for deposition of a filter.

TABLE 1

Layer	Thickness (Å)	Flowrate (SCCM) Ar	Flow-rate (SCCM) N_2	Flowrate (SCCM) O_2	Potential (V)	Power (kW)	Pressure (μ)	No. Passes	Substrate Speed (in/min)
TiO_2	327	71	0	131	-371	40	1.5	8	47
NiCr	12	170	0	0	-444	1	3.0	1	154
Ag	100	69	0	0	-552	10	1.5	1	154
NiCr	12	170	0	0	-444	1	3.0	1	154
Si_3N_4	461	12	60	0	-387	15(x2)	5.0	2	31

The above filter had the following optical and electrical characteristics:

82.4 % Transmittance (integrated D65 source)
6.1 % Reflectance of the film covered side
11.5 % Absorbance
10.5 Ω/ square Electrical sheet resistance
0.09 Emissivity

The durability of the inventive filter of Table 1 was tested. The procedures of the chemical and mechanical tests that were performed are described in Table 2. The inventive filter passed all the tests.

Curve 1 in Fig. 1b illustrates the reflectance of the interference filter produced under the parameters set forth in Table 1 as from the film side. Curve 3 is the reflectance of the uncoated substrate side and curve 5 is the transmittance. The measurements were performed with a scanning spectrophotometer.

TABLE 2

Test Conditions and Scoring Procedures

1.	Humidity Test	Exposures in a humidity cabinet for: (1) 24 hrs. at 90/C and 98% RH and (2) 96 hrs. at 60/C and 98% RH.
2.	Salt Fog Test	20% Salt Fog, 95-98/F for 72 hrs.
3.	UV Exposure Test	Exposure for 24 hrs. with cycles of 4 hrs. condensation until failure or 120 hrs.
4.	Ammonium Test	Samples are placed upright in closed container of 50% ammonium hydroxide solution at room temperature for 5 hrs.
5.	Salt Dot Test	A 1% salt solution is applied to a filter paper dot placed on the film with the sample placed in a constant humidity environment for 24 hrs.

Evaluations of the above tests are based on both microscopic evaluation and emissivity measurements. The details of the evaluations are:

A. Samples are scored for evidence of microscopic corrosion as seen under 200x magnification on a scale of 1 to 10, where 10 is unaffected and 1 is completely corroded.

B. Measure the change in emissivity due to corrosion. The scoring is based on:

Emissivity Score = 10 (Emiss. before/Emiss. after)

C. Recorded scores are an average of 1 and 2

6.	Taber Abrasion	Samples are subjected to a total of 50 revolutions on the Taber abrader, using the standard 500 gram weight and CS-10F wheels.

Evaluation is based on the average number of scratches seen under 50x magnification in 4 $inch^2$ areas. Using the equation below gives a score of 0 for more than 55 scratches in a 1" square area and 10 for none:

$$\text{Taber Score} = 10 - [(\text{No. scratches}) \times (0.18)]$$

As stated above, filters of the invention are those in which one or both of the dielectric layers comprise a composite film of either SiZrN, SiTiN, SiHfN, or mixtures thereof. For each composite, the relative amount of silicon nitride ranges from approximately 60-95% by weight depending on whether the composite is used as the first or second dielectric layer. The index of refraction of the composite film correspondingly ranges from approximately 2.4 (60% silicon nitride) to approximately 2.05 (95% silicon nitride).

One method of depositing composite films is cosputtering of a cylindrical magnetron employing dual targets with one target being made of silicon and the other target being made of either zirconium, titanium, hafnium, or mixtures thereof. When cosputtering with dual cathodes with nitrogen as the reactant gas, the angle of the magnetic assembly of each target can be adjusted to get homogeneous composition distribution. See PCT Patent Publication No. WO 92/01081 and Belkind et al., "Reactive Co-Sputtering of Oxides and Nitrides using a C-MAGT Rotatable Cylindrical Cathode," Surface and Coating Technology, 49 (1991), 155-160.

Another method of depositing composite films is to have one or more alloy targets, each coated with silicon and either zirconium, titanium, hafnium, or a mixture thereof. A process for fabricating cylindrical alloy targets involves doping silicon and another metal (or other metals) to form a conductive silicide. For instance, doping silicon and zirconium results in forming $ZrSi_2$, a conductive silicide that possesses a bulk resistivity of approximately 160 micro ohm cm. This material is conductive enough to be sputtered by a magnetron. The silicide can be synthesised by heating zirconium and silicon together (hot press technique) to a sufficient temperature to form $ZrSi_2$. Thereafter, the silicide is grounded to a powder and sprayed onto a stainless steel backing tube to form a homogeneous coating.

ZrSiN composite films were formed by co-sputtering a C-MAG rotatable magnetron system manufactured by Airco Coating Technology. The system employed dual cathode targets wherein the angle the magnetic assembly of each target was set at approximately 45° relative to normal so as to focus the ZrN and Si_3N_4 molecules onto the glass substrates. It is believed that ZrN is the predominant form of zirconium nitride created in the sputtering process, although other forms may be produced as well. Thus, unless otherwise stated, ZrN will represent all forms of zirconium nitride sputtered.

With dual targets, the relative amounts of reactively sputtered material deposited from each target can be regulated, in part, by adjusting the power to each target. Employing this technique, three different ZrSiN composite films were deposited. The first film comprised of approximately 60% Si_3N_4 and 40% ZrN (60/40), the second comprised of approximately 72% Si_3N_4 and 28% ZrN, and the third comprised of approximately 83% Si_3N_4 and 17% ZrN (83/17).

Curves 30 and 32 in Fig. 3 illustrate the percentage transmission in the visible light region for films one (60/40) and three (83/17), respectively; curves 40 and 42 in Fig. 4 illustrate the percentage reflection in the visible light region for films one (60/40) and three (83/17), respectively; and curves 50 and 52 in Fig. 5 illustrate the percentage absorption for films one (60/40) and three (83/17), respectively.

Table 3 sets forth the refractive index (n) and extinction coefficient (k) values versus wavelength (λ) for the first composite film (60% Si_3N_4, 40% ZrN), and Table 4 sets forth the optical values versus wavelength for the second composite film (72% Si_3N_4, 28% ZrN). (The optical values were measured by an ellipsometer.)

TABLE 3

λ	n	k
380	2.600	0.0500
400	2.566	0.0500
420	2.557	0.0400
440	2.542	0.0350
460	2.521	0.0300
480	2.500	0.0250
500	2.472	0.0200
520	2.463	0.0150
540	2.449	0.0150
560	2.436	0.0150
580	2.424	0.0100
600	2.412	0.0110
620	2.404	0.0090
640	2.396	0.0080
660	2.389	0.0070
680	2.382	0.0060
700	2.376	0.0060
720	2.371	0.0060
740	2.366	0.0060
760	2.361	0.0050
780	2.356	0.0040
800	2.353	0.0030
820	2.349	0.0030
840	2.347	0.0001
860	2.344	0.0000
880	2.341	0.0000
900	2.338	0.0000
920	2.337	0.0000
940	2.335	0.0000
960	2.332	0.0000
980	2.332	0.0000
1000	2.329	0.0000
2000	2.300	0.0000

TABLE 4

λ	n	k
300	2.4972	0.1768
350	2.3298	0.0718
400	2.2752	0.0400
450	2.2298	0.0156
500	2.2122	0.0071
550	2.1957	0.0001
600	2.1886	0.0028
650	2.1813	0.0051
700	2.1779	0.0060
800	2.1724	0.0070
1000	2.1673	0.0070
2000	2.1500	0.0070

As is apparent, refractive index in the visible region was higher for the first composite film which has less Si_3N_4.

Claims

1. A thin film interference filter comprising:
   a transparent substrate;
   a first dielectric layer;
   a first metal precoat layer;
   an optically reflective metal layer;
   a second metal precoat layer; and
   a second dielectric layer
   in which one or both of the dielectric layers is a composite comprising silicon nitride and one or more of zirconium nitride, titanium nitride, and hafnium nitride or mixtures thereof.

2. A thin film interference filter according to Claim 1 in which one or both of the dielectric layers is a composite comprising of silicon nitride and zirconium nitride.

3. A thin film interference filter according to Claim 1 or Claim 2 in which at least one of the dielectric layers comprises approximately 60 to 95% by weight of silicon nitride.

4. A thin film interference filter according to any preceding claim in which the other dielectric layer comprises silicon nitride.

5. A thin film interference filter according to any preceding claim in which the other dielectric layer is also composite and comprises silicon nitride and one or more of zirconium nitride, titanium nitride, and hafnium nitride.

6. A thin film interference filter according to Claim 5 in which the other dielectric layer comprises silicon nitride and zirconium nitride.

7. A thin film interference filter according to Claim 5 or Claim 6 in which the other dielectric layer comprises approximately 60 to 95% by weight of silicon nitride.

8. A thin film interference filter according to any preceding claim in which the composite dielectric layer has

a thickness of about 200 to 500 Å.

9. A thin film interference filter according to any preceding claim in which the other dielectric layer has a thickness of about 300 to 500 Å, preferably 350 to 500 Å.

10. A thin film interference filter according to any preceding claim in which one or both metal precoat layer is formed from a metal selected from one or more of nickel, chromium, tungsten, and platinum or alloys thereof and in which the optically reflective metal layer is formed from one or more of silver, gold, copper, and platinum or alloys thereof.

11. A thin film interference filter according to Claim 10 in which one or both precoat layer is a metal film wherein the metal elements comprise approximately 80 to 95 weight % nickel and 5 to 20 weight % chromium.

12. A method for the production of a durable thin film interference filter on a transparent substrate, with the filter having a substantially neutral visible reflected colour, comprising the steps, in sequence, of:
reactively sputtering a first substantially transparent dielectric layer having a refractive index of approximately 2.0 to 2.7 onto the substrate;
depositing a first metal precoat layer;
depositing a partially reflective metal layer;
depositing a second metal precoat layers and
reactively sputtering a second substantially transparent protective dielectric layer onto said metal precoat layer.

13. A method of producing a durable interference filter according to Claim 12 in which the step of reactively sputtering the second dielectric layer comprises the steps of:
providing a cylindrical magnetron having a silicon coated rotatable target and having magnetic means disposed at an angle of approximately 30° to 80° from normal relative to the substrate; and
moving the substrate towards the rotatable target so that dielectric material reactively sputtered from the target is focused unto the substrate as it approaches the target.

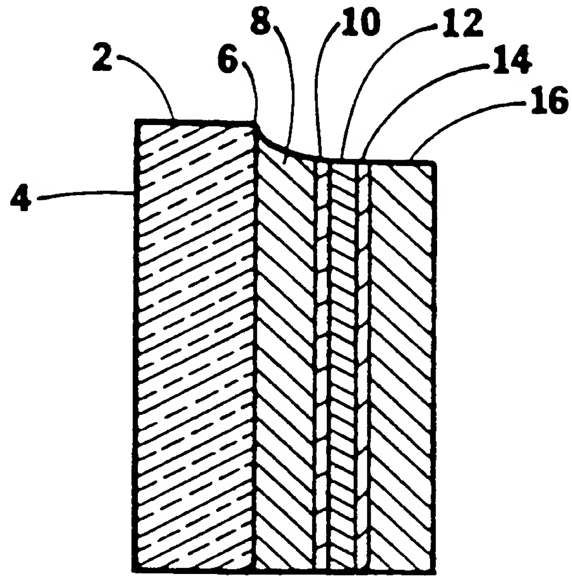
2
4
6
8
10
12
14
16

FIG._1A.

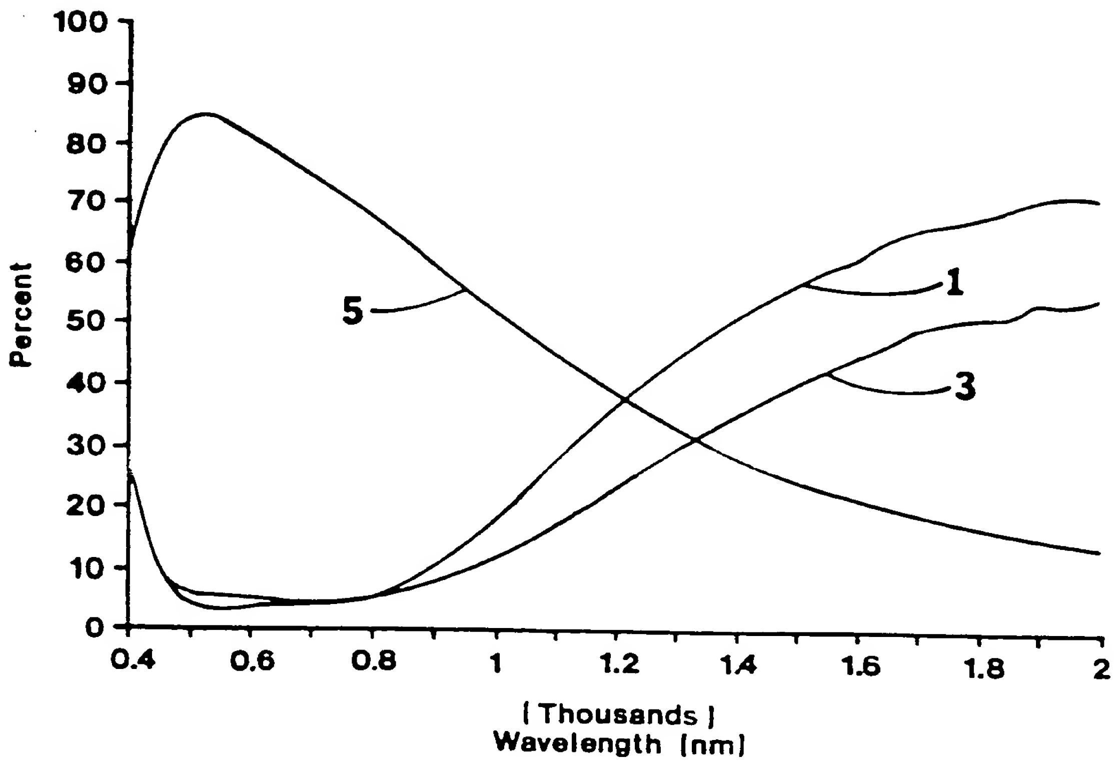
100
90
80
70
60
50
40
30
20
10
0
Percent
5
1
3
0.4
0.6
0.8
1
1.2
1.4
1.6
1.8
2
(Thousands)
Wavelength (nm)

FIG._1B.

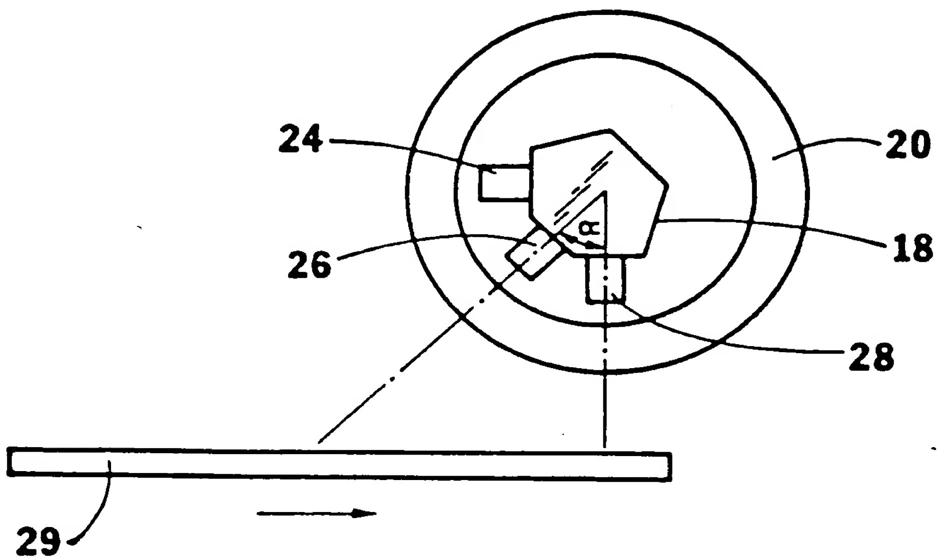
24
20
26
18
28
29
α

FIG._2.

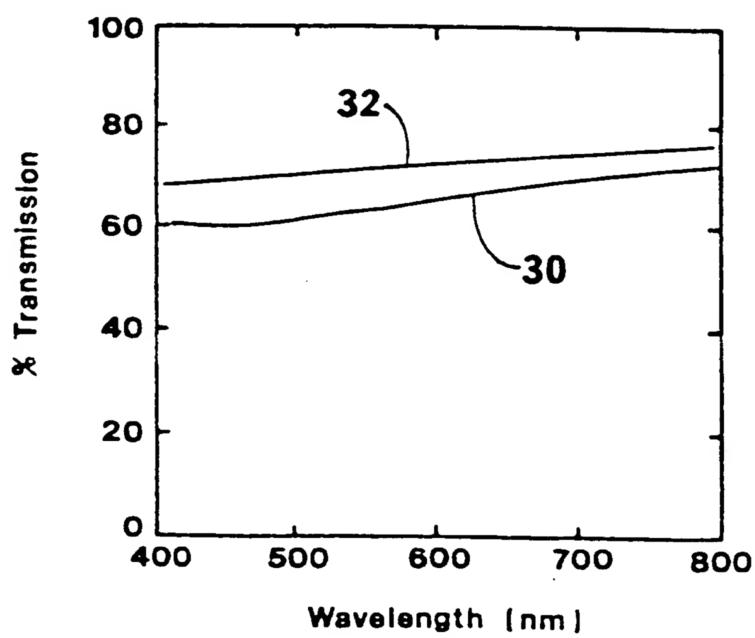
100
80
60
40
20
0
400
500
600
700
800
% Transmission
Wavelength (nm)
32
30

FIG._3.

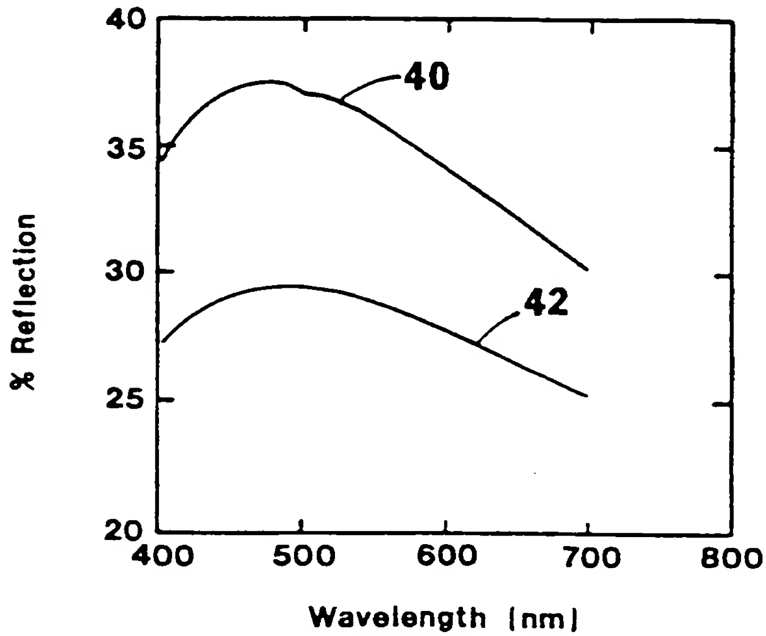
40
35
30
25
20
400
500
600
700
800
% Reflection
Wavelength (nm)
40
42

FIG._4.

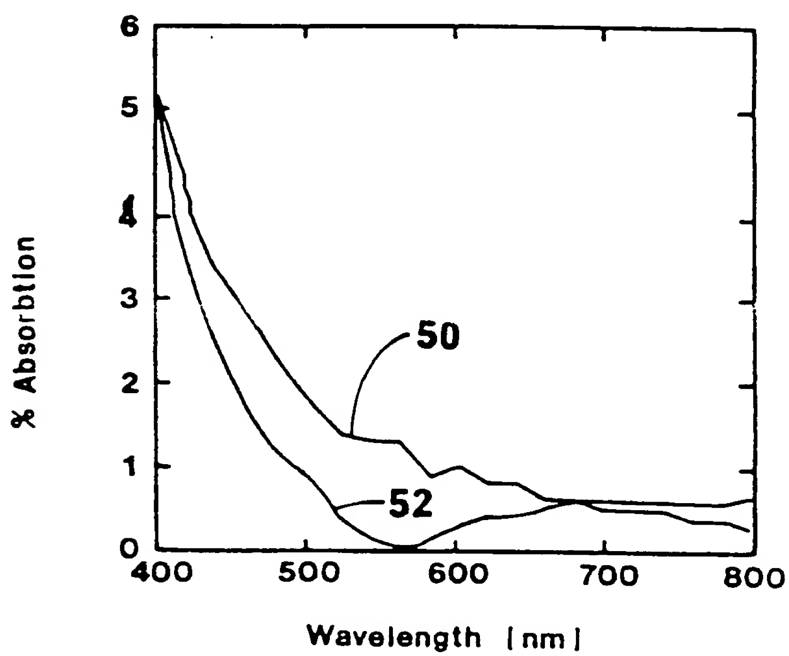
6
5
4
3
2
1
0
400
500
600
700
800
% Absorbtion
Wavelength (nm)
50
52

FIG._5.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 93 30 1635

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
Y A	EP-A-0 456 487 (BOC GROUP) * the whole document *	1,2,5,6,10 3,4,7,11-13	G02B5/28 C03C17/36
Y	PATENT ABSTRACTS OF JAPAN vol. 13, no. 049 (C-565)3 February 1989 & JP-A-63 242 948 (ASAHI GLASS) 7 October 1988 * abstract *	1,2,5,6,10	
A	PATENT ABSTRACTS OF JAPAN vol. 12, no. 338 (C-527)12 September 1988 & JP-A-63 100 043 (NIPPON SHEET) 2 May 1988 * abstract * & DATABASE WPIL Week 2388, Derwent Publications Ltd., London, GB; AN 88-158891 & JP-A-63 100 043 (NIPPON SHEET) 2 May 1988 * abstract *	8-11	TECHNICAL FIELDS SEARCHED (Int. Cl.5) G02B C03C
A	US-A-5 047 131 (BOC GROUP) * column 2, line 60 - column 3, line 31 * * column 2, line 55 - column 6, line 57; figure 1 *	12,13	

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	01 JULY 1993	LIPP G.

CATEGORY OF CITED DOCUMENTS

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